

# **Final Technical Report**

## **Large Area Lateral Epitaxial Overgrowth (LEO) of Gallium Nitride (GaN) Thin Films on Silicon Substrates and Their Characterization**

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# PENDEO-EPITAXIAL GROWTH OF GAN ON SIC AND SILICON SUBSTRATES VIA METALORGANIC CHEMICAL VAPOR DEPOSITION

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## ABSTRACT

Pendeo-epitaxial lateral growth (PE) of GaN epilayers on (0001) 6H-silicon carbide and (111) Si substrates has been achieved. Growth on the latter substrate was accomplished through the use of a 3C-SiC transition layer. The coalesced PE GaN epilayers were characterized using scanning electron diffraction, x-ray diffraction and photoluminescence spectroscopy. The regions of lateral growth exhibited  $\sim 0.2^\circ$  crystallographic tilt relative to the seed layer. The GaN seed and PE epilayers grown on the 3C-SiC/Si substrates exhibited comparable optical characteristics to the GaN seed and PE grown on 6H-SiC substrates. The near band-edge emission of the GaN/3C-SiC/Si seed was 3.450 eV (FWHM  $\sim 19$  meV) and the GaN/6H-SiC seed was 3.466 eV (FWHM  $\sim 4$  meV).

## INTRODUCTION

Prior to the advent of the lateral growth techniques, heteroepitaxial gallium nitride films typically contained threading defect densities that exceeded  $10^9/\text{cm}^2$ . These defects seriously compromised the properties of subsequently fabricated devices. New growth techniques leading to low defect-density nitride films were required to achieve commercialization of viable optoelectronic and microelectronic devices.

The advancement of lateral epitaxial overgrowth (LEO) as a technique capable of producing GaN epilayers with defect densities reduced to  $10^5/\text{cm}^2$  has recently been demonstrated by several research groups.<sup>1,2,3,4</sup> The significance of lateral overgrowth was immediate as Nichia Chemical reported in 1997 that use of LEO aided in increasing the lifetime of their blue laser diode from a few hundred hours to over 10,000 hrs,<sup>5</sup> and then subsequently introduced the first commercially available GaN blue laser diode.<sup>6</sup> However, to benefit from this reduction in defects, the placement of devices incorporating LEO technology is confined to regions on the final GaN device layer that are located over the masked regions and not over the window regions of the GaN seed layer. Figure 1 shows a scanning electron microscopy (SEM) cross-sectional image of GaN grown using the LEO technique.

The authors are currently investigating another lateral growth technique for GaN, namely, pendeo-epitaxy,<sup>7,8,9,10,11</sup> as a process route to resolve the aforementioned alignment problem. Pendeo-epitaxy (from the Latin: *pendeo*—to hang, or to be suspended) incorporates mechanisms of growth used by the conventional LEO process by using masks to prevent vertical propagation of threading defects, and extends the phenomenon to employ the substrate as a *pseudo-mask*. Pendeo-epitaxy (PE) differs from conventional LEO in that growth does not initiate through

open windows but begins on sidewalls etched into the GaN seed layer. As the lateral growth from the sidewalls continues, vertical growth of GaN begins and results in the eventual lateral overgrowth of the masked seed form. Pendeo-epitaxial growth ultimately results in coalescence over and between seed forms, producing a continuous layer of GaN, as shown in Figure 2.

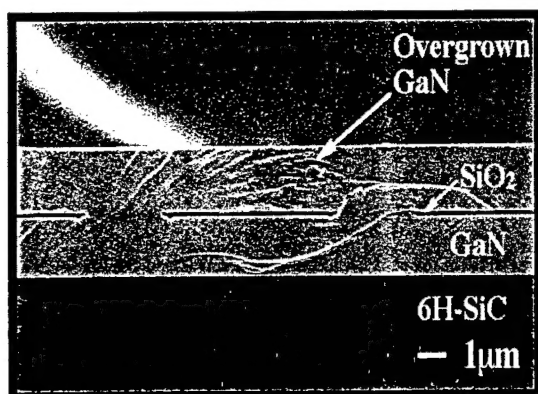


Figure 1. Cross-sectional SEM micrograph of LEO GaN on SiC.

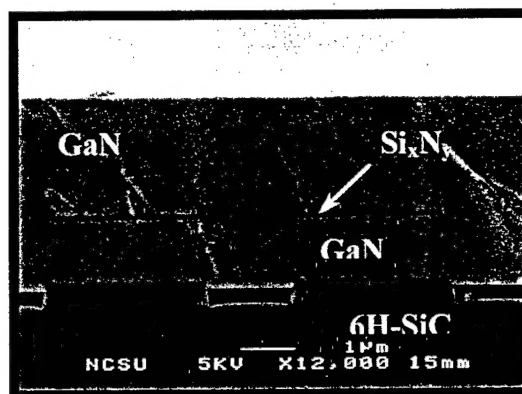


Figure 2. Cross-sectional SEM micrograph of PE GaN on SiC.

Although both LEO and PE research have led to low defect-density GaN material on two-inch SiC and sapphire substrates, the use of this substrate does not resolve the problem of achieving low-cost, large area GaN films necessary for commercialization of microelectronic devices. Meeting this objective has renewed interest in using Si(111) substrates as an alternative to SiC and sapphire. Several recent demonstrations of lateral growth process routes achieving growth of (0001)GaN on (111)Si have been reported.<sup>8,12,13</sup> In this paper we report on the process steps for and characterization of PE GaN epilayers grown on both (0001) 6H-SiC and (111) Si substrates.

## EXPERIMENTAL PROCEDURES

### *GaN Seed Layers*

(0001) 6H-SiC on-axis substrates: The initial 500nm thick GaN seed layers were grown on 100nm thick high-temperature AlN buffer layers previously deposited on the substrates via metalorganic vapor phase epitaxy (MOVPE), as detailed in Ref. 14.

(111)Silicon substrates: 500nm thick (111)3C-SiC transition layers were initially grown on very thin (111)3C-SiC layers produced by conversion of Si substrate surfaces via reaction with C<sub>3</sub>H<sub>8</sub> entrained in H<sub>2</sub>. Both the conversion step and SiC film deposition were achieved using atmospheric pressure chemical vapor deposition (APCVD), as detailed in Ref. 15. The 500nm thick GaN seed layers were subsequently deposited on 100 nm thick AlN buffer layers in the manner used for the 6H-SiC substrates as noted above.

### *Pendeo-epitaxial growth of GaN*

A 100 nm silicon nitride growth mask was deposited on the seed layers via plasma enhanced CVD. A 150 nm nickel etch mask was subsequently deposited using e-beam

evaporation. Patterning of the nickel mask layer was achieved using standard photolithography techniques and Ar-plasma sputtering. The final, tailored microstructure consisting of silicon nitride masked GaN seed forms was fabricated via inductively coupled plasma (ICP) etching of portions of the silicon nitride growth mask, the GaN seed layer and the AlN buffer layer. The seed-forms used for this study were raised rectangular stripes oriented along the  $\langle 1100 \rangle$  direction. Various seed form widths and separation distances were employed. Pendeo-epitaxial growth of GaN was achieved within the temperature range of 1050-1100°C and a total pressure of 45 Torr. The precursors (flow rates) of triethylgallium (26.1  $\mu\text{mol/min}$ ) and  $\text{NH}_3$  (1500 sccm) were used in combination with a  $\text{H}_2$  diluent (3000 sccm). Additional experimental details regarding the pendeo-epitaxial growth of GaN and  $\text{Al}_x\text{Ga}_{1-x}\text{N}$  layers employing 6H-SiC substrates are given in Refs. 7-11.

The morphology and defect microstructures have been investigated using scanning electron microscopy (SEM) (JEOL 6400 FE), transmission electron microscopy (TEM) (TOPCON 0002B, 200KV) and X-ray diffraction (XRD) (Philips X'Pert MRD X-ray diffractometer). Optical characterization was performed using a He-Cd laser ( $\lambda=325\text{ nm}$ ).

## RESULTS AND DISCUSSION

### GaN Seed Layers

The pendeo-epitaxial phenomenon is made possible by using growth and mask mechanisms similar to conventional LEO techniques and by using the substrate itself as a *pseudo*-mask, i.e. the GaN does not nucleate on the exposed SiC surface when higher growth temperatures are employed to enhance lateral growth. The Ga- and N-containing species more likely either diffuse along the surface or evaporate (rather than having sufficient time to form GaN nuclei) from this substrate. Since the newly deposited laterally grown GaN is suspended above the SiC substrate, there are no defects associated with the mismatches in lattice parameters between the PE GaN and the SiC substrate.

Pendeo-epitaxial techniques can be applied in general to other substrates as long as they, or a transition layer deposited on the substrate, act similarly as a *pseudo*-mask. As demonstrated above, silicon carbide meets this requirement. Therefore, by using a transition layer of 3C-SiC, silicon can be successfully used as a substrate for PE growth. In addition to acting as a *pseudo*-mask, the 3C-SiC performs two other key functions. Firstly, deposition on (111)Si results in the growth of (111)3C-SiC. The atomic arrangement of the (111) plane is equivalent to the (0001) plane of 6H-SiC; thus it facilitates the sequential deposition of a high temperature (0001) 2H-AlN buffer layer of sufficient quality needed for the GaN seed layer. Secondly, since the AlN buffer does not act as a *pseudo*-mask, it must also be removed from the areas between the GaN seed forms to prevent the undesired nucleation of 'defective' GaN in these areas. Without the presence of an AlN buffer or other transition layer, the silicon substrate is exposed to the growth environment and consequent effects resulting from the reaction of Si atoms with the Ga and N species. Therefore the 3C-SiC transition layer acts as a reaction/diffusion barrier between the Si substrate and the GaN.

Figure 3 shows a SEM micrograph of a 500 nm (111) 3C-SiC transition layer deposited on an on-axis (111)Si substrate. A typical high resolution DCXRD scan is shown in Figure 4. Although the quality of the 3C transition layer is less than optimal, as indicated from the relatively rough morphology visible in Figure 3, and typical XRD FWHM values of  $0.5^\circ$ , it is of sufficient quality for the deposition of 2H-AlN and 2H-GaN seed layers.



The surface morphology of the GaN seed layers deposited on the (111) 3C-SiC transition layers were comparable to GaN seed layers deposited on (0001) 6H-SiC substrates, and had very smooth surfaces. Low temperature (14K) photoluminescence analysis of GaN seed layers grown on both 6H-SiC and 3C-SiC/Si substrates are shown in Figure 5.

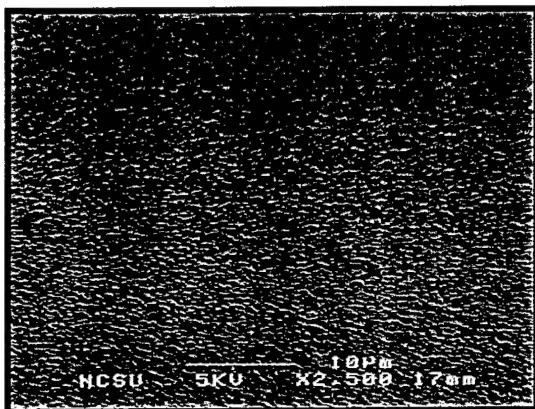


Figure 3. Plane-view SEM of a 0.5 micron thick 3C-SiC transition layer on (111)Si.

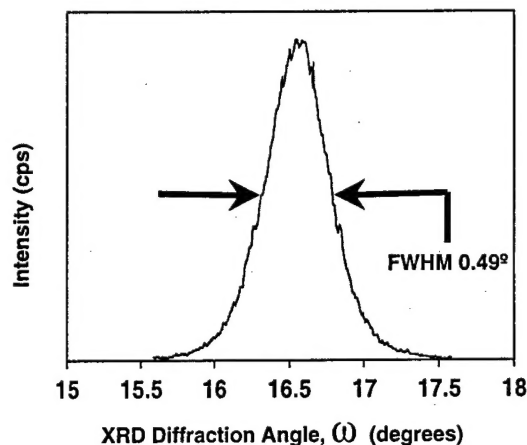


Figure 4. X-ray rocking curve of the 111 diffraction of a 0.5 micron thick 3C-SiC transition layer.

The near band-edge emission was 357.7 nm (3.466 eV, FWHM  $\sim$  4meV) and 359.4 nm (3.450 eV, FWHM  $\sim$  19 meV), respectively, and has been attributed to an exciton bound to a neutral donor ( $X-D^0$ ). These results show that the quality of the GaN seed layers deposited on 3C-SiC/Si substrates is approaching the optical quality of GaN grown on 6H-SiC substrates.

#### *Pendeo-Epitaxial Growth:*

Figure 2 shows an SEM micrograph of a PE grown GaN using a 6H-SiC substrate with a 1 micron thick GaN seed layer and a 100nm thick silicon nitride mask. The seed microstructure was comprised of 3  $\mu$ m wide posts and 1.5  $\mu$ m wide trenches oriented in the  $\langle 1100 \rangle$  direction. Analysis of Figure 2 reveals coalescence between and above the seed structures resulted forming a continuous epilayer of GaN.

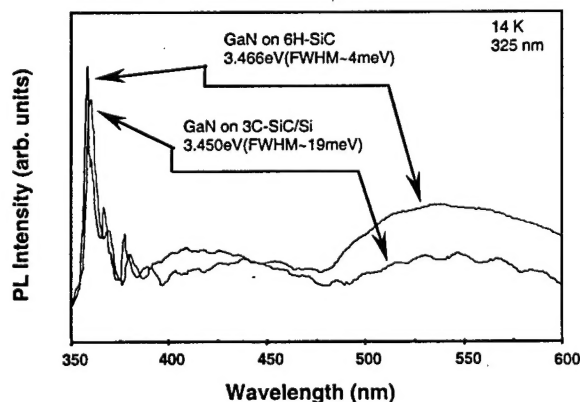


Figure 5. Low temperature (14 K) PL spectra of GaN seed layers grown on 6H-SiC and 3C-SiC/Si substrates.

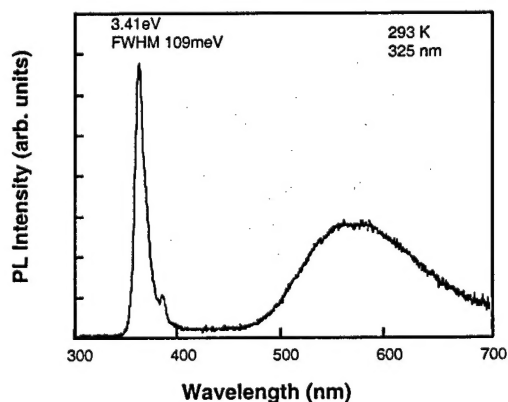


Figure 6. Room temperature PL spectrum of coalesced PE GaN on a 3C-SiC/Si substrate.

Figure 6 shows the room temperature PL spectrum for a coalesced GaN PE epilayer grown on a 3C-SiC/Si substrate. A band-edge emission of 363.6 nm (3.41 eV) was observed and indicates the PE GaN on silicon is under tensile stress.

Figures 7 and 8 show SEM micrographs of PE grown GaN using 3C-SiC/Si substrates. The seed microstructures were 2  $\mu\text{m}$  wide posts and 3  $\mu\text{m}$  wide trenches oriented in the  $\langle 11\bar{0}0 \rangle$  direction. The sample shown in Figure 7 employed a 200 nm silicon nitride mask on top of the seed forms. Analysis of this sample revealed void formation and poor coalescence over the 'thick' seed mask. Void formation in the trench regions is also visible; however this does not appear to effect the quality of the coalesced GaN above the trench. This is typical of PE GaN grown under nonoptimized conditions. The sample in Figure 8 is a maskless PE GaN epilayer. PE GaN grown using this seed microstructure configuration is the equivalent of LEO techniques, such that vertical propagation of threading defects from the original GaN seed is not prevented. Analysis of Figure 8 reveals the same void formation at the coalescence point in the trench regions.

Although the cause of these voids is not yet fully understood, it is believed that optimization of the initial seed microstructure, including post-trench ratio, seed thickness, etch quality and mask thickness, result in their elimination. Preliminary results suggest that minimization of the trench width reduces the void formation in the trench region. Also, minimizing the seed mask thickness helps to eliminate void formation in and poor coalescence of GaN grown over the mask. Research optimizing all of the process and fabrication parameters is ongoing.

Figure 9 shows a typical X-ray rocking curve for a PE GaN sample grown on a 3C-SiC/Si substrate. Similar to the case of GaN grown using LEO techniques, there is  $\sim 0.2^\circ$  tilting of the laterally grown GaN compared to that of the seed layer, in the  $\langle 11\bar{2}0 \rangle$  direction (i.e. perpendicular to the seed form orientation). However, unlike the LEO technique, it is difficult to determine if the PE material, the LEO material, or both are tilted since there is both pendeo-epitaxial growth between and LEO growth above the original seed forms. Work is in progress to make this determination.

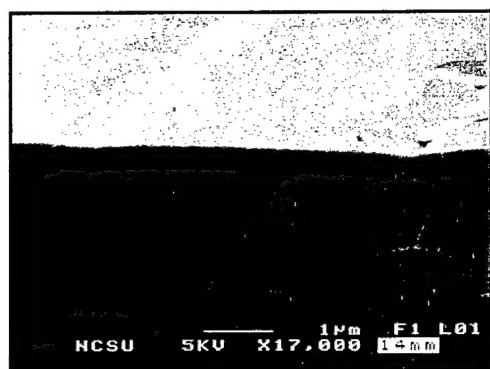


Figure 7. 45° SEM view of PE GaN grown on a 3C-SiC/Si substrate.

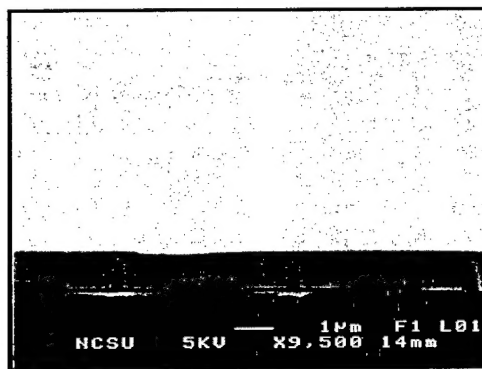


Figure 8. 45° SEM view of maskless PE GaN grown on a 3C-SiC/Si substrate.



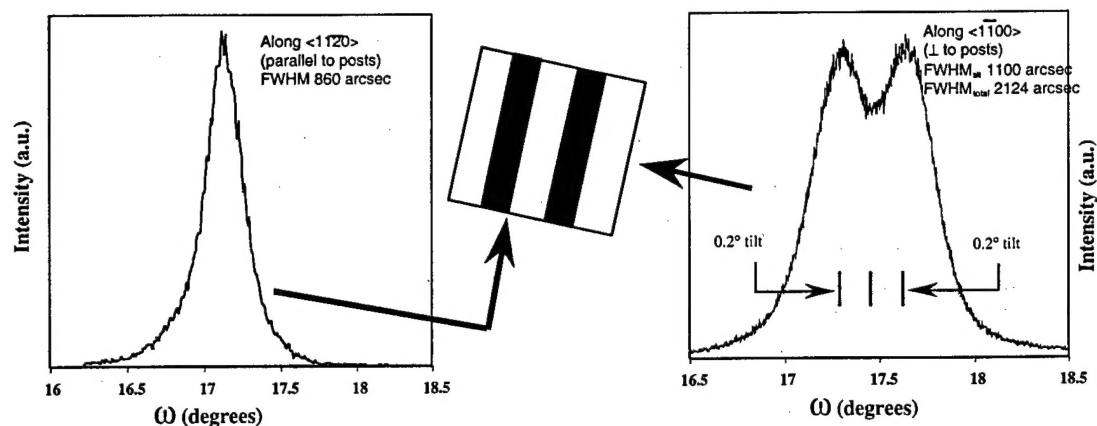


Figure 9. DCXRD rocking curves of the 0002 diffraction of PE GaN grown on 3C-SiC/Si substrates.

## CONCLUSIONS

The pendeo-epitaxy process route has been developed as an alternative to the conventional GaN LEO technique and as a means of confining all the vertically threading defects, stemming from the GaN/AlN and AlN/SiC interfaces, within the seed forms. This results in the growth of a more uniform low defect-density GaN epilayer. Incorporation of silicon nitride masks and SiC *pseudo*-masks (either as the 6H-SiC substrate or a 3C-SiC transition layer) combined with etched sidewalls of GaN seed forms has allowed the achievement of PE GaN films with low dislocation densities over the entire GaN epilayer surface. The quality of GaN seed layers grown on 3C-SiC/Si substrates was shown to be comparable to GaN layers grown on 6H-SiC. Investigations regarding the optimization of the PE growth technique, including determination of the ideal GaN seed thickness, ideal seed microstructure geometry (e.g. post width, trench width, etc.) and ideal seed mask material, is ongoing. Additionally, optimization of fabrication steps for the PE GaN seed forms (e.g., photolithography, mask alignment, ICP etching process, etc.) is underway.

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